U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

AD-A025 484

HIGH VELOCITY BEHAVIOR OF DISLOCATIONS IN COPPER

CALIFORNIA INSTITUTE OF TECHNOLOGY

PREPARED FOR

ARMY RESEARCH OFFICE

30 APRIL 1976

Unclassified

168080 ARO 11007.3-MC

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

ADA025484

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ARO-11007.3-MC	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) HIGH VELOCITY BEHAVIOR OF DISLOCATIONS IN COPPER		5. TYPE OF REPORT & PERIOD COVERED Final Report: 1 Oct 1972 - 31 Mar 1976
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) T. Vreeland, Jr. David S. Wood		8. CONTRACT OR GRANT NUMBER(s) DA-ARO-D-31-124-73-G47 DAHC04 75 G 0027
9. PERFORMING ORGANIZATION NAME AND ADDRESS California Institute of Technology Pasadena, California91109		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office Post Office Box 12211 Research Triangle Park, NC 27709		12. REPORT DATE 30 Apr 76
		13. NUMBER OF PAGES 18
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Crystal growth
Dislocations
Velocity
Torsion
Copper
lasers

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Dislocation velocity measurements were attempted using torsion pulses of 10μ sec duration to move dislocations at near-sonic velocities in copper single crystals. It was concluded that the torsion pulse technique was not applicable to the study of near sonic dislocation velocities in currently available test crystals. Efforts were then directed toward:(a) the gorwth of more perfect copper crystals (with a considerably increased initial dislocation spacing), and (b) the development of a new loading system, designed to produce high amplitude stress pulses of shorter

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

Abstract continued,

duration (1/4 μ sec) than obtainable with the torsion pulse technique (about 5 μ sec).

The new loading system employs a giant laser pulse, and is briefly described in this report. The crystal growth system developed for the production of highly perfect copper crystals is also described, together with the results obtained to date on the perfection of the crystals grown with the new system.

"HIGH VELOCITY BEHAVIOR OF DISLOCATIONS IN COPPER"

FINAL REPORT

BY T. VREELAND, JR. and DAVID S. WOOD

APRIL 30, 1976

U.S. ARMY RESEARCH OFFICE - DURHAM

GRANT NUMBERS:

DA-ARO-D-31-124-73-G47

DAHC04-75-G-0027

CALIFORNIA INSTITUTE OF TECHNOLOGY

PASADENA, CALIFORNIA

ABSTRACT

Dislocation velocity measurements were attempted using torsion pulses of 10 μsec duration to move dislocations at near-sonic velocities in copper single crystals. Displacements of individual dislocations could not be determined because dislocation displacements were larger than the initial dislocation spacing, making it impossible to follow the path of an individual dislocation. Observation of the rate of growth of slip bands, which was used to indicate dislocation velocities at lower stresses, was unsuccessful because individual bands were not observed in the crystals subjected to higher stresses.

It was concluded that the torsion pulse technique was not applicable to the study of near sonic dislocation velocities in currently available test crystals. Efforts were then directed toward: (a) the growth of more perfect copper crystals (with a considerably increased initial dislocation spacing), and (b) the development of a new loading system, designed to produce high amplitude stress pulses of shorter duration (1/4 μsec) than obtainable with the torsion pulse technique (about 5 μ sec).

The new loading system employs a giant laser pulse, and is briefly described in this report. The crystal growth system developed for the production of highly perfect copper crystals is also described, together with the results obtained to date on the perfection of the crystals grown with the new system.

1. Introduction

The torsion pulse testing system, developed by Pope, et al,[1] was used to measure dislocation mobility in copper by Greenman, et al,[2] and Jassby and Vreeland.[3,4,5] Dislocation velocities up to about 90 m/sec were observed in these measurements, and the velocity increased linearly with resolved shear stress (up to about 1.7 M Pa). The velocity increased with decreasing temperature at constant stress, and it was concluded that phonon drag was the primary source of resistance to dislocation motion at resolved stresses greater than about 0.1 M Pa.

The behavior of dislocations at much higher stress levels, such as those encountered in shock wave loading, has been the subject of much speculation. No direct measurements of dislocation velocity or dislocation drag have been made at high stresses, and the few theoretical treatments of the behavior of dislocations at speeds near the elastic shear wave velocity (2.14×10^3 m/sec for a [111] wave in copper) have not been tested.

The work reported here was undertaken to explore the high velocity behavior of dislocations in copper.

2. Stress Pulse Experiments

A practical lower limit to the stress pulse duration in the torsion testing system is 5 μsec, since the rise time of the stress pulse is about 2 μsec. A dislocation moving at 2×10^3 m/sec is displaced 10 mm in 5 μsec. It is impractical to follow the displacement of an individual dislocation when the displacement exceeds the initial dislocation spacing. Since this spacing is less than 10 mm in the best copper single crystals available, we hoped to observe the growth of slip bands rather than the displacement of individual dislocations in the stress pulse experiments.

Torsion pulses at higher stress levels than those reported previously[5] were found to produce general slip with considerable dislocation multiplication. This is in contrast to the slip band formation observed at the lower stress levels. This observation led us to conclude that crystals with very low initial dislocation density would be required for successful stress pulse tests at the higher stress levels. Effort to produce such crystals was intensified and is reported in Section 3 of this report. Crystals with an initial dislocation spacing of a few millimeters could be used for the high velocity measurements if the stress pulse duration could be reduced by an order of magnitude. A system for producing a single compression pulse of about $1/4\,\mu$sec was developed, and is described in Section 5 of this report. Development of a system for strain measurement at 4.2°K is described in Section 2, and dislocation observations are discussed in Section 4.

2.1 Low Temperature Strain Measurement

Torsion pulse tests with a given torsional impulse will produce the highest dislocation velocity at the lowest test temperature. For this reason, we used a test temperature of 4.2°K. Earlier tests at 4.2°K indicated a difficulty with the doped silicon piezoresistive strain gages used to monitor the stress pulse. The gage resistance became very high when they were cooled below about 38°K. The problem was avoided by locating the gages away from the cold part of the torsion rod where the test specimen was attached. This solution was not satisfactory for short duration stress pulse tests, because it necessitated subtracting a large portion of the torsional impulse measured by the gages in order to obtain the relatively small impulse applied to the specimen. Suitable gages for use at 4.2°K were developed.

The silicon strain gages with p-type dopant levels near 10^{18} acceptors/cm^3 act as resistors above about 38°K. The rapid rise in resistance below 38°K, indicating the electrons were freezing out of the conduction band, suggested the need for higher doping levels. At a doping level greater than 10^{19} acceptors/cm,^{3} the silicon should act as a pure resistor rather than a semiconductor. If the piezoresistive output at 4.2°K is adequate, gages with the higher doping levels should be useable.

Gage sets with doping levels of 3×10^{19} and 1.4×10^{20} acceptors/cm^3 were obtained from Micro Gage, Inc., of El Monte, Calif. Both sets of gages are useable at 4.2°K. Gages with the higher dopant level have been calibrated, and were found to have slightly greater output at 4.2°K than at room temperature. The room temperature gage factor is reduced by about 30% when the doping level is increased from 10^{18} to 1.4×10^{20} acceptors/cm^3. (The reduced output causes no problems in the torsion testing experiments).

2.2 Compression Stress Pulse Tests of Zinc

When it became apparent that our copper crystals were not suitable for stress pulse testing at high stress levels, experimental efforts were diverted to compression tests to determine the velocity and drag force on second order pyramidal dislocations in zinc. Techniques were avilable to measure these parameters for both screw and edge dislocations, and high compressive stress levels were readily obtainable to explore the high velocity behavior.

A compressional stress pulse loading system based on a Hopkinson bar technique was employed for measurement of the mobility of pyramidal edge and screw dislocations in zinc at room temperature and at 77°K. The rate of slip band growth was deduced from the experiments, and equated to

location velocity. Resolved shear stresses from 10 M Pa to 80 M Pa were employed, and dislocation velocities from 20 m/sec to nearly 600 m/sec were measured. Dislocation damping coefficients obtained from these measurements and from measurements on the basal system are given in Table I.

TABLE I

Values of the Dislocation Damping Coefficients for Edge and Screw Dislocation in Zinc in Units of 10^{-5} Pa sec

Temperature °K	Basal System		Second Order Pyramidal System	
	Screw	Edge	Screw	Edge
300	3.4(6)	3.5(6)	16	27
77		1.2(7)	7.1	11

The damping coefficient for edge dislocations in the basal system increases by a factor of 2.9 between 77°K and room temperature. The factor is 2.4 and 2.3 for pyramidal edge and screw dislocations respectively.

The theoretical treatment of dislocation damping by Al'shits and Indembom[8] identifies the phonon processes which contribute to the damping as strain field scattering and phonon relaxation (phonon viscosity). Their expression for these contributions involves parameters characteristic of the third order modulus for the dislocation and the phonon spectrum and strain field near the dislocation core. These parameters are not known for dislocations in zinc; however, the temperature dependence of the drag coefficients for each of the processes is different. An increase by a factor

of 2.1 between 77°K and room temperature is predicted for the phonon relaxation process, while a factor of 5.8 is predicted for the phonon scattering process. Our data indicates that phonon relaxation (phonon viscosity) provides the major contribution to dislocation damping in zinc between 77°K and 320°K.

Attempts to explore the near sonic velocity behavior using the Hopkinson bar technique were unsuccessful because dislocation multiplication rapidly increased at higher stresses. Slip bands were no longer found, and the resulting plastic strain considerably perturbed the stress pulse amplitude and duration.

3. Crystal Growth

Crystals grown by the Bridgeman method were used in the experiments of this investigation. In order to obtain test specimens of a higher perfection than those obtainable from these crystals, a Czochralski system similar to that reported by Sworn and Brown (J. Crystal Growth, 12, 195 (1972) was assembled. The system employs a purified graphite crucible with a 35 mm inside diameter. An atmosphere of purified argon is used, and the graphite crucible is heated by means of an induction coil which surrounds the crucible, its two tubular mullite radiation shields, and two water-cooled glass tubes (water flows axially through a 2 mm annulus between the inner quartz tube and the outer Pyrex tube). Radiation shields on the crystal pull rod and on the bottom of the graphite crucible limit axial heat loss from the system. Four thermocouples monitor the temperature of the graphite mold. Two thermocouples are 10 mm below the center of the charge, and the other two are placed about 10 and 40 mm above the top of the charge on the outside of the graphite mold.

The crucible is fixed while the crystal pull rod is driven in rotation as well as up or down along the axis of the crucible. The crucible and heat shields each have a pair of holes located slightly above the molten charge level through which the seed crystal and crystal growth are observed.

3.2 Crystal Growth

We have developed experience in pulling Cu crystals, and have successfully pulled crystals up to about 15 mm diameter. A constant rate of pulling is used, and the temperature in the graphite mold, just below the center of the charge, is held constant to within ± 1/2°C. This temperature control is accomplished by a servo system in which the necessarily very small input power variations are made by varying the position of a parasitic R.F. pickup coil relative to the main induction coil. Varying the position of the parisitic coil varies the inductive coupling between it and the main coil, causing the power dissipated in a resistive load connected to the parasitic coil to change so as to maintain a constant temperature. Rather abrupt changes in diameter were produced when very small, manual changes in the power supplied to the main induction coil were made. With the use of the parasitic coil to control the temperature, abrupt diameter changes are avoided.

Strain-free removal of the as-grown crystals is accomplished by casting paraffin wax around the top and bottom of the crystal before the small diameter neck is cut to separate the crystal from the seed. These wax castings are joined by another wax casting to prepare the crystal for acid sawing.

4. Dislocation Observations

An asymmetrical topographic camera, reported by Kuriyama, Early, and Burdette (AAA 12th Aerospace Sciences Meeting, Washington, D.C., paper 74-204, 1972) was assembled and used. This camera is capable of detecting crystal substructure with angular misorientations on the order of 5". Crystals grown by the Bridgeman method show angular misorientations considerably in excess of 5", while the Czochralski-grown crystals show no substructure. Dislocation densities of the grown and annealed crystals are being measured by etch pit techniques. Preliminary results on the as-grown crystals indicate a density equal to that of the best Bridgeman grown crystals after annealing (about 10 mm^{-2}). Annealing typically lowers the dislocation density of the Bridgeman crystals by about one order of magnitude.

5. Pulse Loading Systems

Stress pulses of less than one μ sec duration may be produced by the impact of a thin flyer plate on the test specimen or by the laser pulse loading system which we have developed. The laser pulse system has the following advantages over the flyer plate system:

1. Eliminates the very critical angular alignment condition of the flyer plate system. The flyer plate must be parallel to specimen surface within less than 10^{-3} radian in order that the stress wave front be within 0.1 radian from the specimen surface.

2. Requires only one optically-flat surface, on the back side of the specimen, rather than optically-flat surfaces front and back.

3. Permits control of the pressure distribution over the surface of the specimen so that unwanted radial stress waves may be controlled.

4. Simplified recovery of the specimen without producing uncontrolled dislocation displacements.

5.1 The Laser Pulse System

A method for the generation of a single short-duration stress pulse in a solid was described in our Interim Technical Report No. 2, under Contract No. DA-ARO-D-31-124-73-G47, submitted 1 May 1975. This method employs a giant laser pulse of 10 to 20 nanosec duration to heat a layer of liquid on top of the solid. The heating occurs at constant volume, producing a pressure which is relieved by a wave propagating through the liquid layer.

We have completed the design and construction of the test fixture for the laser pulse loading system.

5.2 The Text Fixture

The fixture for laser pulse loading is shown in Fig. 1. The horizontal laser beam, LB, enters at the upper left. It is turned into a vertical direction by the 45 degree mirror M. and its diameter is adjusted by lens L_1 to match that of the test specimen, TS.

The test specimen is 1 cm in diameter and about 1 mm thick. A thin layer of liquid on the surface of the test specimen absorbs the laser pulse energy and applies pressure to the specimen for a time proportional to the thickness of the liquid layer. A liquid layer 0.25 mm thick produces a pressure pulse of about 0.25 μsec duration.

The test specimen rests upon a thin diaphragm, which is clamped under the locating plate, LP. This diaphragm serves to retain the liquid and to prevent the specimen from falling after the stress wave has passed through it, and the momentum trap, MT, and quartz gage, QG, have moved downward.

The momentum trap and quatz gage are held in position under the specimen, diaphragm, and locating plate by a small force provided by spring plate SP acting upward through the energy absorber, EA. The expendable energy absorber

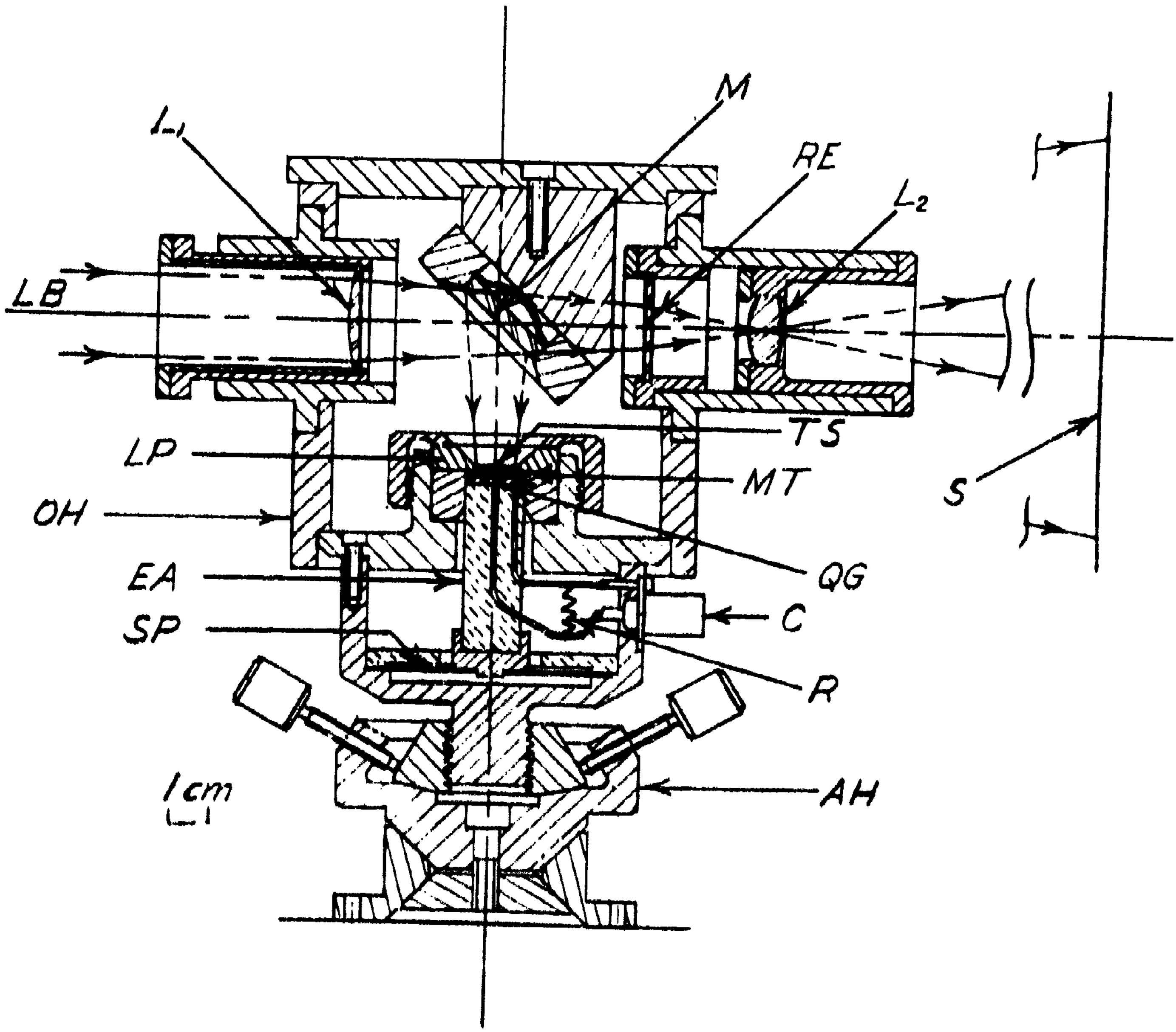

Fig. 1

Laser Pulse Loading Fixture, Cross-sectional View

is made of polyurethane foam so that it absorbs the recoil kinetic energy of the momentum trap and quartz gage by crushing.

Electrical leads from the quartz gage are routed through holes and slots in the energy absorber to the shunt resistor, R, and coaxial cable Connector C. The signal is displayed and photographed on a suitable oscilloscope to provide the measurement of the stress time history.

The optical head, OH, at the top of the apparatus incorporates means for observing the alignment with respect to the laser beam while the laser is operated in a continuous low intensity manner. The mirror, M, is removed so that the laser beam illuminates the graduated glass reticle, RE. The distance from the intersection of the vertical and horizontal optical axes to the test specimen and reticle are the same so that the size and location of the laser beam spot on the test specimen and reticle are the same. Lens L_2 focuses an enlarged image of the reticle and laser beam spot on the screen, S, where they may be observed while alignment adjustments are made.

The alignment head, AH, at the base of the apparatus, provides the means of leveling the test specimen and aligning the apparatus with respect to the incident laser beam. A spherical seat with four adjustment screws provides the leveling adjustment which is necessary to secure uniform thickness of the liquid layer. The optical head is removed and a spherical bubble level placed on the specimen mount during this operation. The spherical seat also provides for adjustment of the angular orientation of the apparatus about the vertical axis. The screw thread employed to attach the spherical seat to the upper portion of the apparatus provides the means to adjust the vertical position of the horizontal optic axis. A cross-slide below the spherical seat permits adjustment in the horizontal direction transverse to the axis of the incident laser beam.

The test fixture has been built and is ready for testing.

5.3 The Radial Release Wave Gage

Stress waves are generated at the cylindrical boundary of the specimen during the period when a uniaxial strain wave is propagating through the thickness of the specimen. These radial release waves must be controlled so that they do not produce significant dislocation displacements. The amplitude of these waves near the center of the specimen is of particular interest and concern because both the amplitude and duration are maximum at the center.

The radial release waves are to be studied by use of a thin flat circular disc of (111) oriented silicon in place of the specimen crystal. The silicon contains an integral piezoresistive element (formed by diffusing boron into the n-type silicon), which measures both the uniaxial wave propagated through the thickness of the disc and the subsequent radial release waves near the center of the disc.

Silicon wafers have been prepared for the boron diffusion, and silicon momentum traps to be used with the gages have been prepared. These silicon discs were ground optically flat and then cut to the appropriate diameter.

The magnitude and duration of the radial release waves will be measured in experiments in which the intensity of laser beam is modified in various ways in the region near the periphery of the specimen. Thus a tailoring of the special distribution of energy in the laser beam can be determined such that the radial release waves will not produce spurious dislocation movement.

5.4 Laser Pulse Testing

Preliminary laser pulse tests will be made during the next several months, using a 2 Joule capacity laser. Dr. Albert Ellis of the University of California at San Diego is making his laser facility available for these

tests. A 25 Joule laser at the Lawrence Radiation Laboratory (LR), Livermore, will be used for the laser pulse tests during the summer of 1976. Prof. D. S. Wood will spend two months at LRL as a visiting consultant performing the laser pulse tests on Cu crystals. Crystal preparation will be carried out at the California Institute of Technology, and the crystals will be transported to LRL by automobile to assure their arrival in a damage-free condition. Tested crystals will be returned to C.I.T. by auto for dislocation observations.

6. Technical Personnel

The following technical personnel worked on the project:

T. Vreeland, Jr. - Professor of Materials Science

David S. Wood - Professor of Materials Science

Kenneth M. Jassby - Research Fellow in Materials Science

Gregory P. Hamill - Graduate Research Assistant

Alvin Illig - Sr. Lab. Technician

Henri J. Arnal - Sr. Technician

Kenneth R. Elliott - Summer Graduate Research Assistant

Lawrence Lichtmann - Undergraduate Lab. Assistant

Jeffrey J. Harrow - Undergraduate Lab. Assistant

Glenn R. Ierley - Undergraduate Lab. Assistant

Curtis Meissner - Undergraduate Lab. Assistant

Charles E. Kistler - Undergraduate Lab. Assistant

None of the personnel involved received an advanced degree earned by them while employed on the project.

7. Technical Reports and Publications

Interim Technical Report No. 1 (Grant No. DA-ARO-D-31-124-73-G47):

K. M. Jassby and T. Vreeland, Jr., "Mobility of Fast-Moving Second-Order Second-Order Pyramidal Dislocations in Zinc at 77°K and 296°K".

Interim Technical Report No. 2 (Grant No. DA-ARO-D-31-124-73-G47):

D. S. Wood and T. Vreeland, Jr., "An Experiment to Produce and Measure High Dislocation Velocities Using a Laser Pulse Loading Technique".

Papers Submitted (Grant No. DAHC04-75-G-0027):

K. M. Jassby and T. Vreeland, Jr., "Investigation of Pyramidal Edge and Screw Dislocation Mobility in Zinc by a Compressional Stress Pulse Technique", submitted to Mat. Sci. and Eng.

K. M. Jassby and T. Vreeland, Jr., "Mobility of Pyramidal Edge and Screw Dislocations in Zinc at 77°K", submitted to Scripta Met.

REFERENCES

1. D. P. Pope, T. Vreeland, Jr., and D. S. Wood, "Machine for Producing Square Torsion Pulses of Microsecond Duration", Rev. Sci. Instr., 35, 1351 (1964).

2. W. F. Greenman, T. Vreeland, Jr., and D. S. Wood, "Dislocation Mobility in Copper", J. Appl. Phys., 38, 3595 (1967).

3. K. M. Jassby and T. Vreeland, Jr., "An Experimental Study of the Mobility of Edge Dislocations in Pure Copper Single Crystals", Phil. Mag., 21, 1147 (1970).

4. K. M. Jassby and T. Vreeland, Jr., "Dislocation Mobility in Copper and Zinc at 44°K", Scripta Met., 5, 1007 (1971).

5. K. M. Jassbv and T. Vreeland, Jr., "Dislocation Mobility in Pure Copper at 4.2°K", Phys. Rev. B., 8, 3537 (1973).

6. D. P. Pope and T. Vreeland, Jr., "Mobility of Basal Dislocations in Zinc", Phil. Mag., 20, 1163 (1969).

7. V. I. Al'Shits and V. L. Indenbom, Sov. Phys. USPEKHI, 18, 1 (1975).